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THE QUENCH HARDENING OF TITANIUM-MANGANESE ALLOYS

Interim Technical Report No. 1

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THE QUENCH HARDENING OF TITANIUM-MANGANESE ALLOYS

Interim Technical Report No. 1

Object: To evaluate the nature of the quench-hardening reaction in Ti-Mn alloys utilizing a critical review of the literature.

Summary: Evaluation of the quench-hardening reaction has been made on the basis of (a) consideration of alloy systems showing a similar crystallographic transformation (b.c.c. to c.p.h.), and (b) consideration of systems showing similarity in phase diagrams to the Ti-Mn diagram.

Conclusions: From a study of the evidence presented, the quenching transformation is believed to be of the martensitic type. The unstable α' formed on quenching, probably with a c.p.h. structure, may be responsible for the quench-hardening phenomenon in the Ti-Mn alloys.

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Introduction

Much work has been done in recent years to improve the strength of titanium-base alloys. Titanium has great potential value as an engineering material because it has one of the highest strength-to-weight ratios of materials known today and, in addition, displays superior corrosion resistance under various operating conditions. Moreover, the existence of an allotropic transformation and the concomitant possibilities for quench hardening have interested research workers engaged in alloy development.

Previous Work on the Ti-Mn System

According to recent work (1)[*], the beta-to-alpha (b.c.c. to c.p.h.) transformation in iodide titanium occurs at 882.5° ± 1°C. It is known that the beta phase in pure titanium cannot be retained even by an extremely rapid quench to room temperature. The same phenomenon is also encountered in other pure metals, such as zirconium, thallium and iron. However, the introduction of alloying elements often makes the transformation of the high temperature phase so sluggish that a portion or all of this phase is retained on quenching or even slow cooling. This behavior is typical of titanium.

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[*] References are listed consecutively at the back of the report

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A number of elements lower the transformation temperature of beta titanium and tend to stabilize this phase. Among these elements are columbium (2), cobalt (3), chromium (4), copper (5), iron (6), manganese (7), molybdenum (2), nickel (8), silicon (9), tantalum (7), vanadium (10), tungsten (7) and zirconium (11).

Addition of the beta-stabilizing elements, manganese and chromium, has produced titanium alloys which quench harden. Figure 1, based on Battelle (12) data, shows the effect of homogenizing temperatures on hardness of titanium alloys containing up to 10% manganese. Greatest hardening is obtained on quenching from 1700°F, at which temperature all alloys used are in the beta field, with alloys containing 3-7% manganese developing maximum hardness. In addition, Table 1 (12) shows the effect of quenching temperatures on the final hardness of manganese and chromium alloys.

Table 1. Effect of Quenching Temperature on
Hardness of Ti-Mn and Ti-Cr Alloys

Composition in %	Vickers Hardness Number Homogenized at 950°C. for 10 min. in argon atmosphere	
	Quenched in cold water (0°C.)	Quenched in liquid nitrogen (-196°C.)
Mn		
1.0	268	218
3.5	377	383
5.0	373	544
10.0	351	351
Cr		
2.5	319	304
3.5	342	368
5.0	332	540
10.0	324	359
15.0	322	314

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From Table 1, the effect of quenching in liquid
nitrogen seems to be similar in both the Ti-Mn and Ti-Cr
systems. Maximum hardness of almost identical value occurs
in the 5% alloy of each system. It is of interest to note
that the 5% alloys are the only ones to show an extensive
increase in hardness between the cold water quench and
liquid nitrogen quench data. Similar changes in hardness,
both positive and negative, were observed in both systems
for manganese or chromium content above and below five
percent.

It is also interesting to note that age hardening
occurs in the Ti-Mn alloys on annealing in the alpha-beta
field and that highest hardness among the alloys listed is
attained in the 5% Mn alloy. This is shown in Fig. 2, (12),
which summarizes the isothermal transformation data obtained
with Ti-Mn alloys homogenized at 950°C for 10 minutes in an
argon atmosphere and quenched into a lead bath at 350 ± 3°C.

The titanium-rich Ti-Mn equilibrium diagram
determined at Battelle (7) is reproduced here as Fig. 3.
The main features of this partial equilibrium diagram are
that the β transus line decreases continuously with increasing
Mn content, the amount of solubility of Mn in α phase is quite
limited, and there is some evidence of the existence of a
eutectoid reaction.

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The Ti-Mn Quench-Hardening Reaction

The occurrence of quench hardening in the Ti-Mn system has been adequately substantiated in the present literature survey. It is the purpose of the present investigation, therefore, to study the conditions under which such hardening occurs and to determine the mechanism underlying that behavior.

The transformation in Ti-Mn alloys can be considered on the basis of (a) structural alteration (from b.c.c. to c.p.h.), and (b) the similarity existing between the Ti-Mn phase diagram and other alloy systems.

(a) Consideration of the b.c.c. to c.p.h. transformation:

Not many alloy systems show transformations like the beta-to-alpha transformation in titanium. The most extensively studied system was the Cu-Al system first investigated by Wassermann (13) in the composition range from 11.9 to 12.7% Al. Figure 4 shows the copper-rich portion of the Cu-Al equilibrium diagram containing the eutectoid composition. The high temperature phase, β, transforms into a structure of $\alpha + \delta$ on slow cooling, but when rapidly cooled, transforms to β'' through a martensitic transformation. The β' phase, determined later (14), shows a distorted c.p.h. structure.

Wasserman suggested that the phase transformation around β eutectoid composition was as follows: upon "rapid" cooling from the β field (Fig. 4),

β (disordered b.c.c.) → β_1 (ordered b.c.c.) → β' (distorted c.p.h.)

and upon reheating, $\beta' \to \beta_1 \to \alpha + \delta \to \beta$;

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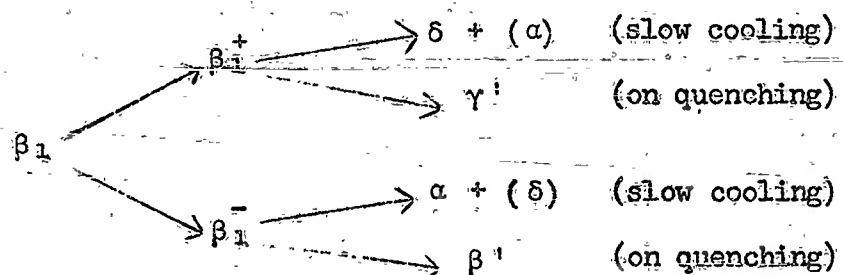
and that the $\beta_1 \rightleftarrows \beta'$ transformation was completely reversible.
Later, Kurdjumow and Stelletzku (15) discovered a new
martensitic lattice having a crystal structure different from
that of β' and which they designated γ'. According to them,
β' resulted from rapid cooling of alloys containing between
12.9 and 13.1% Al, and γ' only from alloys above 13.1% Al.
Microscopic work showed the metallographic structure of β'
to be almost identical with γ', the only difference being
that γ' "needles" often contained apparent twin bands.

Klier and Grymko (16) recently made a microstructural
study of this transformation and clarified the sequence of the
decomposition of β_1 (or β). The isothermal transformation,
at or below eutectoid temperature, in the 13.5% Al specimens
investigated showed that two distinct reactions occurred.
The first reaction resulted in the formation of an acicular
precipitate but the structure was not stable, and on further
holding at that temperature, was subject to a second reaction
of agglomeration and growth. The resulting structure underwent
no further change during continued isothermal holding.

After quenching from the isothermal anneal, prior
to the start of the first reaction, the structure as quenched
was γ' with possible traces of β'. For longer annealing
times, through the course of the first reaction, the structure
in the quenched specimen became a mixture of γ' and β'. This

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meant that the γ' revealed was not original matrix γ' but

rather, because of the formation of the large amount of β',

the γ' formed was of necessity rich in Al. The γ' was next

replaced by δ and the structure was then δ and β'. For

the fully transformed alloy, the structure was composed of

α and δ.

Since both β' and γ' formed from β_1 (or β) of

characteristic composition, Klier and Grymko pointed out

that the structures from the first reaction before quenching

consisted of β_1 of two compositions, namely, β_1^+ (Al-rich)

and β_1^- (Al-depleted). When quenched, β_1^+ transformed to γ'

and β_1^- to β'. On slow cooling, β_1^+ became enriched in Al to

the point where it could precipitate as δ. The precipitation

of δ depleted the β_1^- of Al, to the point where α could

precipitate, the final structure then being made up of δ

and α. The sequence was as follows:

$$\beta_1 \begin{cases} \beta_1^+ \begin{cases} \longrightarrow \delta + (\alpha) & \text{(slow cooling)} \\ \longrightarrow \gamma' & \text{(on quenching)} \end{cases} \\ \beta_1^- \begin{cases} \longrightarrow \alpha + (\delta) & \text{(slow cooling)} \\ \longrightarrow \beta' & \text{(on quenching)} \end{cases} \end{cases}$$

As found by Isaitschen, Kaminsky, and Kurdjumow (14),

γ' is a c.p.h. structure with an ordered arrangement of atoms.

β' differs from γ' in that the [0001] direction is at an angle

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of 2° to the (0001) plane normal, and the angle between
the plane (10$\bar{1}$0) and (01$\bar{1}$0) differs about 1° from 120°.
The writers also indicated that β' can be transformed to
γ' by mechanical deformation, stating that "........after
deformation, some lines on the Debye pattern of β' phase
merge together, giving broad lines; the new line system
becomes analogous to the line system of the γ' phase".

The orientation relationship between β_1 and γ' as
found by Greninger (17), may be defined crystallographically
as follows:

$$(0001)_{\gamma'} \quad \| \quad (110)_{\beta_1} \quad \Big\} \quad A_1$$
$$[01\bar{1}0]_{\gamma'} \quad \| \quad [111]_{\beta_1}$$

$$(0001)_{\gamma'} \quad : \quad (011)_{\beta_1} = 4° \quad \Big\} \quad A_2$$
$$[01\bar{1}0]_{\gamma'} \quad \| \quad [111]_{\beta_1}$$

A_1 and A_2 here are in twin relationship, with the (10$\bar{1}$1)
plane of A_1 as the twinning plane. Since β' is of a pseudo-
hexagonal framework, as mentioned previously, its orientation
relationship (17) can only be approximately represented as:

$$(0001)_{\beta'} \quad : \quad (110)_{\beta_1} = 4°$$
$$[10\bar{1}0]_{\beta'} \quad \| \quad [111]_{\beta_1}$$

In addition, the habit plane as determined by Greninger (17)
is (4$\bar{4}$1)$_{\beta_1}$ for β' and (221)$_{\beta_1}$ for γ' in the transformations

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$\beta_1 \to \beta'$ and $\beta_1 \to \gamma'$ respectively.

The phase transformation in zirconium is another example of the transformation from b.c.c. to c.p.h. structure. From a study of the orientation relationship, Burgers (18) has proposed that the martensite transformation occurs in Zr by a heterogeneous shear on the system (112) [111]. The crystallographic principle underlying his proposal is that the configuration of atoms in the (112) plane of a b.c.c. structure is exactly the same as that in the (10$\bar{1}$0) plane of a c.p.h. structure based on the same atomic radius. Thus a close-packed hexagonal structure can be built up from a b.c.c. structure simply by displacing the (112) planes relative to each other. This mechanism leads to orientations that can be described by the relation:

$$(110)_{b.c.c.} \parallel (0001)_{c.p.h.}$$
$$[111]_{b.c.c.} \parallel [11\bar{2}0]_{c.p.h.}$$

The habit plane resulting from this mechanism would then automatically be the (112) plane. However, the habit plane found by Bowles (19) in the b.c.c. to c.p.h. transformation in Li is a plane bearing Miller indices of the (441) type.

Another possible mechanism presented by Burgers (18) is that the structural transformation from b.c.c. to c.p.h. could have an intermediate f.c.c. transition lattice.

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The structural cycle can be briefly illustrated as follows:

$$b.c.c. \rightarrow f.c.c. \qquad (\beta\text{-brass} \leftrightarrow \alpha\text{-brass})$$

$$(110)_{b.c.c.} \parallel (111)_{f.c.c.}$$

$$[111]_{b.c.c.} \parallel [110]_{f.c.c.}$$

$$f.c.c. \rightarrow c.p.h. \qquad (\beta\text{-Co} \rightarrow \alpha\text{-Co})$$

$$(111)_{f.c.c.} \parallel (0001)_{c.p.h.}$$

$$[110]_{f.c.c.} \parallel [11\bar{2}0]_{c.p.h.}$$

Thus, the net transformation will be:

$$b.c.c. \rightarrow c.p.h.$$

$$(110)_{b.c.c.} \parallel (0001)_{c.p.h.}$$

$$[111]_{b.c.c.} \parallel [11\bar{2}0]_{c.p.h.}$$

which is the same as was just given for the case of Zr.

Weerts (20) reported that the epsilon phase transformation in the Ag-Zn system also had a b.c.c. to h.c.p. structural alteration with the following relationships:

$$(111)_{b.c.c.} \parallel (0001)_{c.p.h.}$$

or

$$(110)_{b.c.c.} \parallel (10\bar{1}0)_{c.p.h.}$$

Bowles (19) reported that the habit plane of martensitic transformation in a b.c.c. matrix was located

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mostly in the vicinity of some minor planes with indices corresponding to {hhl}. No satisfactory theoretical explanation has been offered for this observation.

(b) Consideration of the similarity between phase diagrams:

The quenching of iron-rich Fe-Ni alloys from the high temperature gamma field (f.c.c. structure), Fig. 5, as first reported by Owen and Sully (21), produced a distorted alpha structure (α'). They observed that, after quenching from 800°C a completely distorted alpha, α', structure was present in alloys of Ni content from 2.96 to 23.32 atomic percent and co-existed with retained gamma in alloys from 27.12 to 30.60 atomic % Ni. The lines in the X-ray diffraction pattern of α' are different from those of α, in that they are much broader, the (310) doublet of α' with Co radiation usually being unresolved and diffuse. The material was obviously not in equilibrium, and the lattice was considerably distorted.

It was this distorted structure (α') which was unfamiliar to Osmond and Cartaud (22) and led them to speculate that a simple eutectoid reaction existed in the Fe-Ni equilibrium diagram. Also, this same distorted structure, α' was designated as martensite by Hanson and Freeman (23) owing to its resemblance to the martensite in carbon steel. Later, Owen (24), based on his observations on meteorites,

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described α' as "....a supersaturated alpha solid solution
of Ni iron in which there will be internal stresses caused
by the distortion of the crystal lattice owing to the excess
of Ni,........ Such material is in an unstable condition,
and if heated to moderate temperatures will separate into
the stable components, kanacite and taenite, as found in
meteorites".

The iron-rich portion of the Fe-Mn equilibrium
diagram has phase boundary lines similar to those in the
Ti-Mn system (Fig. 6). As shown by Troiano and McGuire (25),
the gamma transformation in the Fe-Mn system occurs in almost
the same manner as that in the beta-eutectoid Cu-Al system.
On being quenched to room temperature, the gamma transformed
to α' of the same composition and the amount of α' varied
directly with the amount of gamma originally present. The
α' was also characterized by diffuse lines displaced so as
to indicate an abnormally large lattice parameter. The final
X-ray pattern revealed the presence of two alpha solutions,
namely, equilibrium alpha with sharp lines, and supersaturated
alpha with diffuse lines. Gamma of approximately 3 to 15
atomic percent Mn gave an X-ray diffraction pattern corresponding
to the lines of the supersaturated alpha phase.

Gamma of approximately 13 to 30 atomic percent Mn in
the Fe-Mn system formed the unstable epsilon phase (c.p.h.)

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during quenching. The quenching temperature and specimen
size, apparently influenced the amount of epsilon phase
formation. In general, the higher the quenching temperature
and the larger the specimen, the greater was the amount of
epsilon phase formed. However, under no conditions was
epsilon observed below approximately 10 and above 26 atomic
percent Mn.

The transformation temperatures of $\gamma \to \alpha'$ and
$\gamma \to \varepsilon$ are independent of the cooling rate. The
presence of multiple twin-like structure in ε distinguishes
its microstructure from the α phase.

Both ε and retained γ decompose during cold-rolling.
In alloys of 10.62 to 13.00% Mn, the γ retained after quenching
transformed almost entirely to supersaturated α' with a trace
of ε phase. Alloys of 15.24 to 16.74% Mn showed approximately
equal amounts of only γ and ε after quenching. At an inter-
mediate stage of deformation, only ε with a small amount of
α' was present, while after severe deformation, the Debye
pattern indicated the structure to consist almost entirely of
α' with only traces of ε. The large amount of additional α'
could have been formed only from the decomposition of ε.

Duwez (26) also found the supersaturated alpha
structure, α', in the Ti-Mo system by means of X-ray diffraction
patterns. He found it in Ti-Mo alloys of up to 12% Mo, after

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homogenizing at 980°C for 24 hours and quench: from the
beta field. Only partial transformation occured from about
4 to 12% Mo, while above 12% Mo, the β structure was retained.
Also, although the temperature of the allotropic transformation
in pure Ti decreased progressively from 882°C to about 850°C
with increasing rates of cooling from 4° to 10,000° per sec.,
the cooling rate had no effect on the temperature of trans-
formation of β phase into α' (curve M in Fig. 7). He suggests
the retention of β at room temperature might be explained
on the basis of curve M, Fig. 7, extrapolated to the 12% Mo
ordinate line below room temperature.

In alloys showing a transformed α structure, Duwez
reported that the diffuseness of the lines corresponding to
α' structure did not permit him to determine the lattice
parameter of α' structure with any degree of certainty.
Similar observations have been reported for the Fe-Ni (21)
system. Obviously, no amount of annealing will improve the
diffuse lines of α' introduced during quenching, since
annealing would cause decomposition of the α'.

Hansen and his collaborators (2) were unable to
differentiate between the transformed structure (α') and
isothermal α structure on the basis of X-ray diffraction
patterns. Nevertheless, the equilibrium β phase in the
Ti-Mo system will transform during quenching partially or

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wholly into α' at a given temperature in a two-phase region within definite composition limits. It would be reasonable to expect this metastable α' structure to play an important role in the quench-hardening mechanism.

The alloy systems Fe-Ni, Fe-Mn and Ti-Mo discussed above have the same type of equilibrium diagram at the base-metal end as for the Ti-rich Ti-Mn system. Their quenching products also appear to be related closely by the similarity of their equilibrium diagrams and this view can be even extended to the eutectoid-type equilibrium diagrams of Cu-Al (β-eutectoid) and Fe-C systems. Although the structural transformation in the Fe-Ni and Fe-Mn systems is from f.c.c. to b.c.c., the change in the Ti-Mn system is from b.c.c. to c.p.h. The Ti-Mo system, however, which exhibits the same structural change as does the Ti-Mn system, also shows the same quenching product appearing in the Cu-Al (β-eutectoid) system.

Summary

The nature of martensitic transformation is such that it does not require re-adjustment of the concentration of the alloying ingredients, i.e., it is a diffusionless type of reaction and the extent of the reaction usually is an inverse function of the reaction temperature. The martensitic transformation produces a mestastable transition structure, the formation of which is accompanied by considerable hardening

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of the metal. From the evidence presented, the quenching transformation in the Ti-Mn system would seem to be of the martensitic type. However, the form of the transition structure and the possible mechanism by which it is generated will have to be determined experimentally.

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Contract No. DA-30-069-ORD-207
RAD Project No. TB4-10 (DA Project 5-93-08-020)
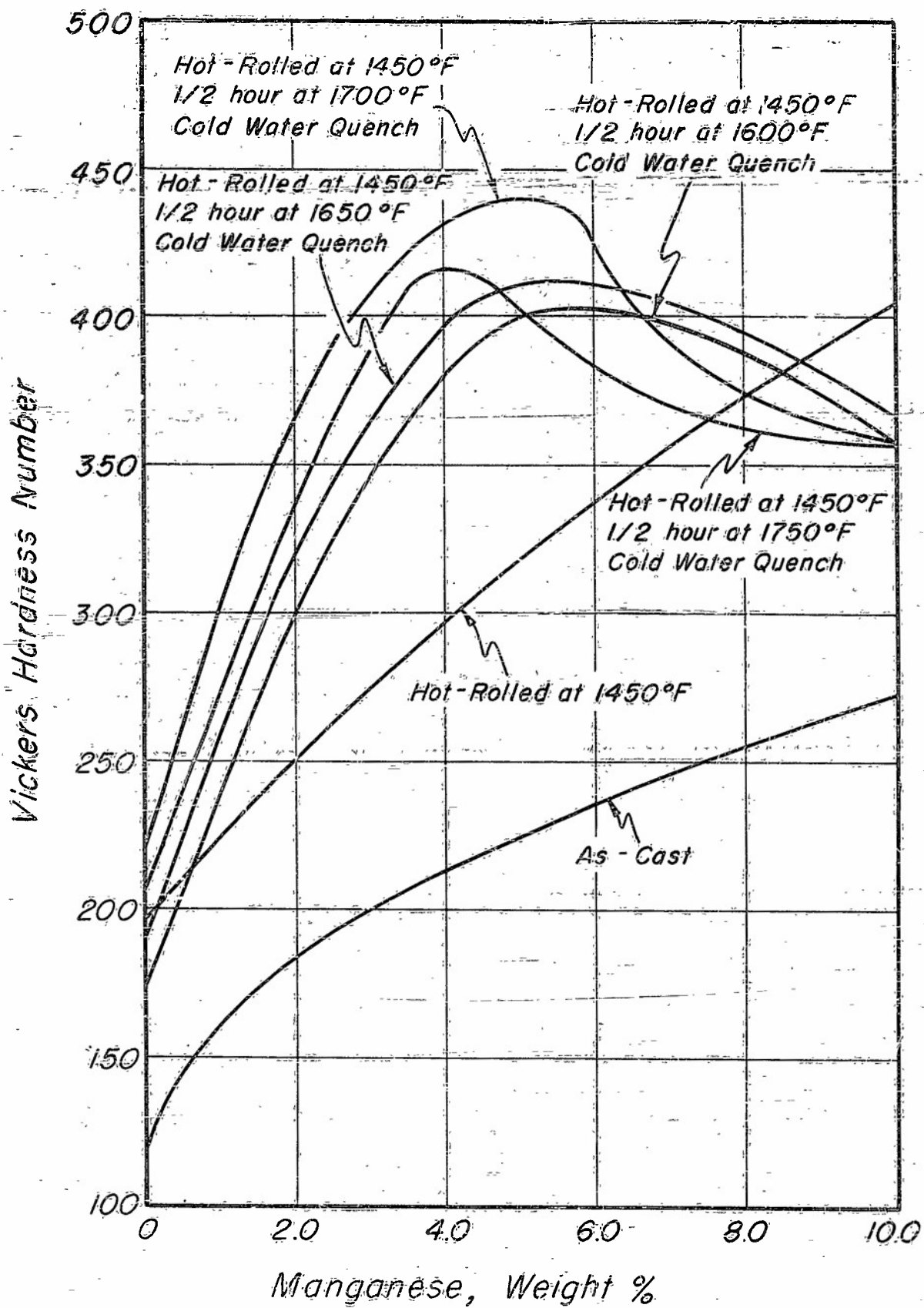RAD No. ORDTB 1-12072

ILLUSTRATIONS

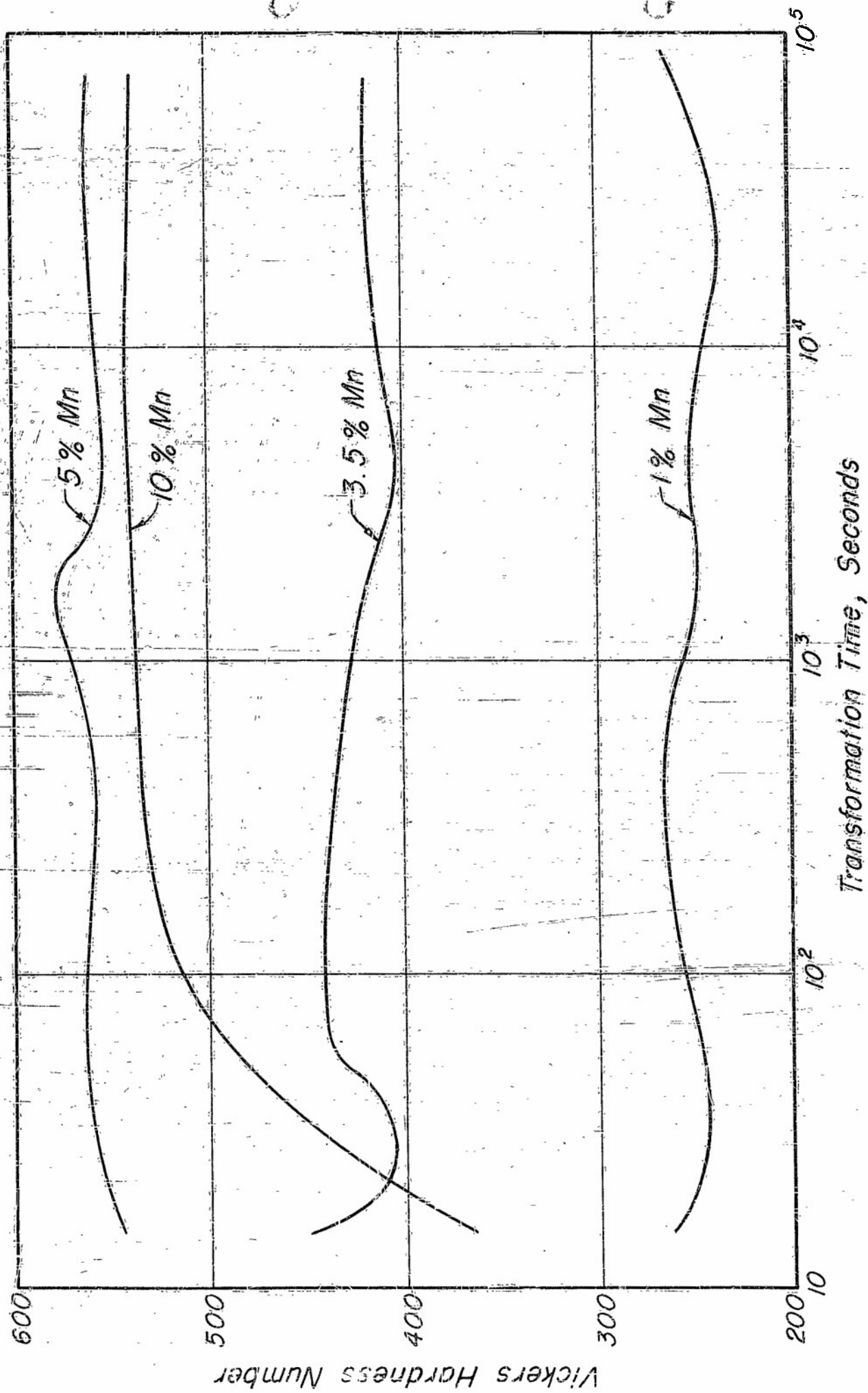Fig. 1 — EFFECT OF HEAT TREATMENT ON Ti - Mn ALLOYS (Battelle)

Fig. 2 — ISOTHERMAL TRANSFORMATION CURVES OF Ti-Mn ALLOYS (Battelle)
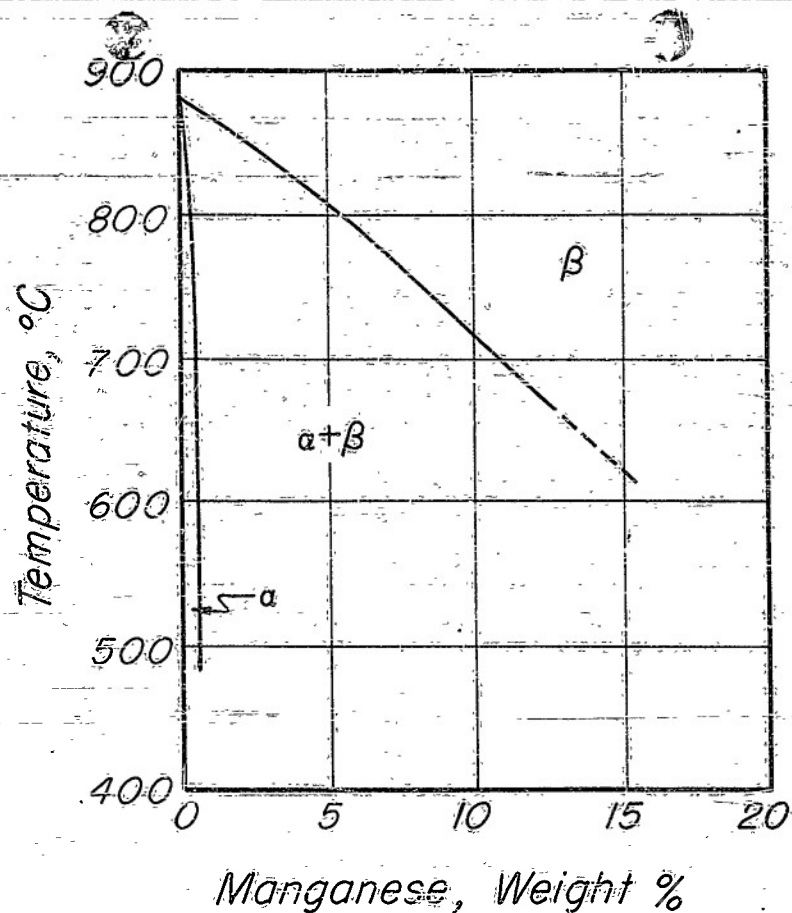
Fig. 3 – Titanium–Rich Portion of the Tentative Ti–Mn
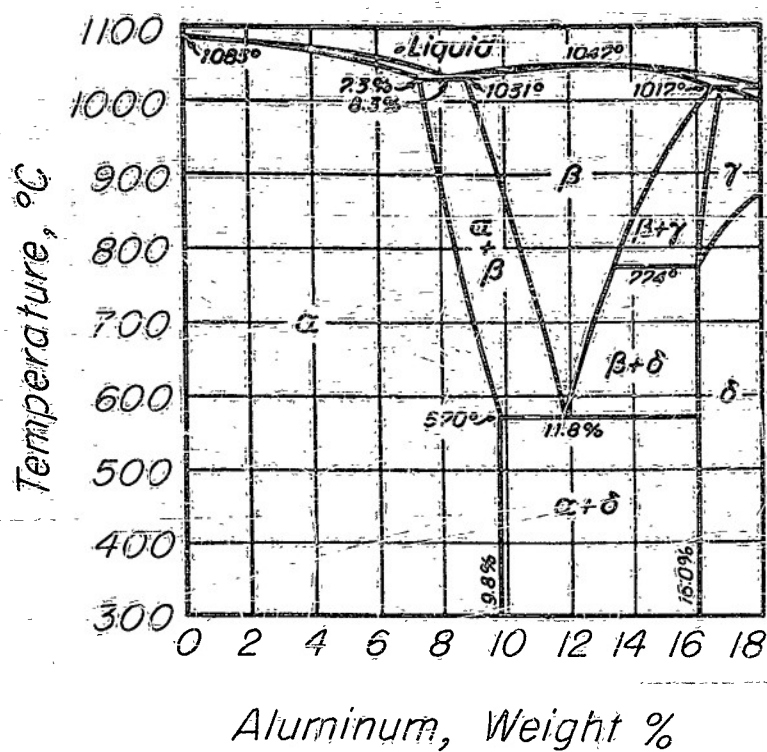Equilibrium Diagram   (Battelle)

Fig. 4 – Copper–Rich Portion of the Cu–Al Equilibrium
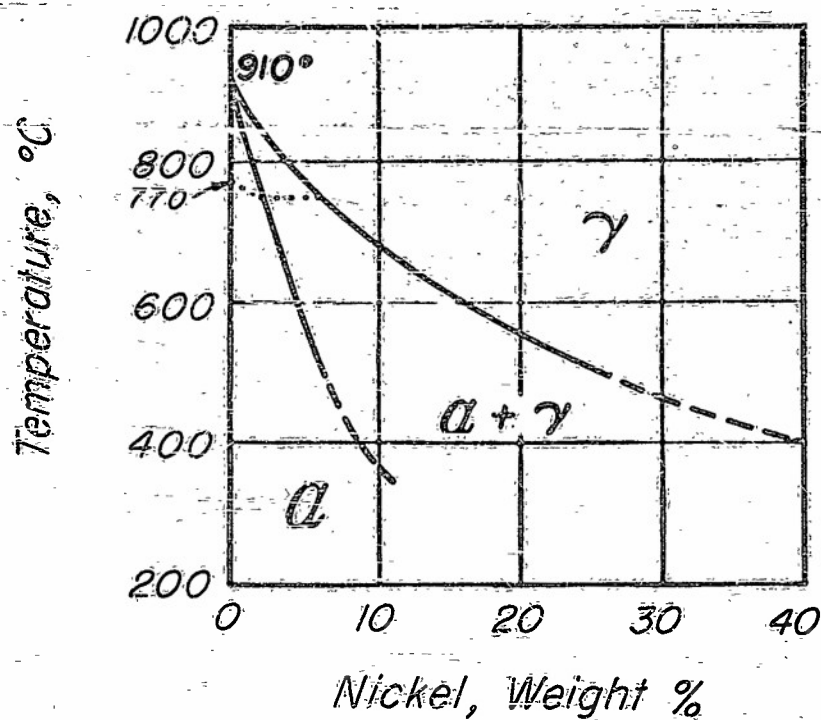Diagram   (National Metals Handbook)

Fig. 5 - Iron - Rich Portion of the Fe - Ni Equilibrium
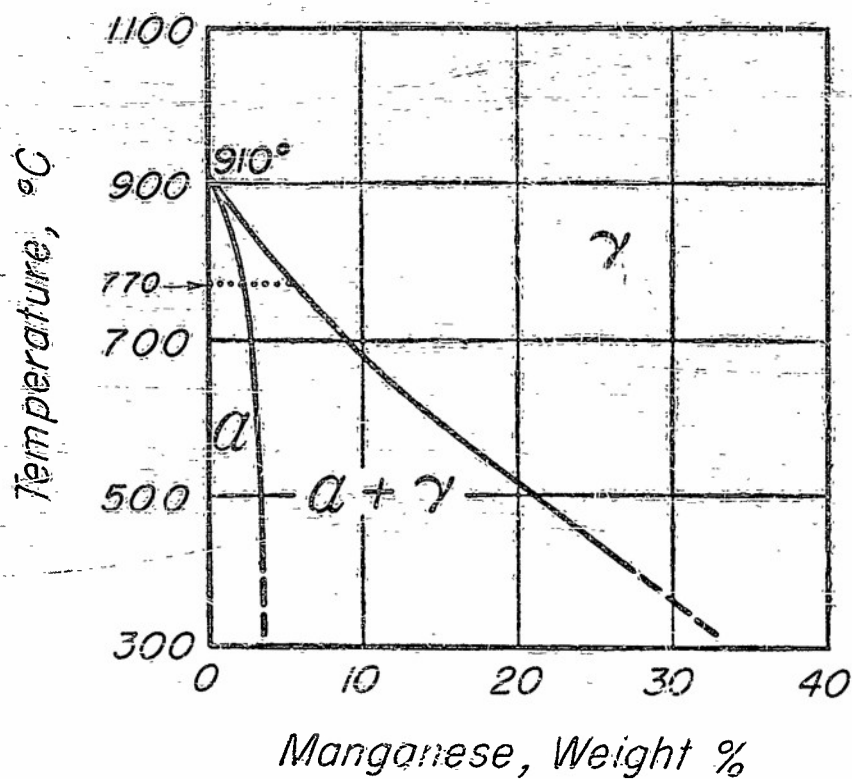Diagram   (Metals Handbook 1948 Edition)

Fig. 6 - Iron - Rich Portion of the Fe - Mn Equilibrium
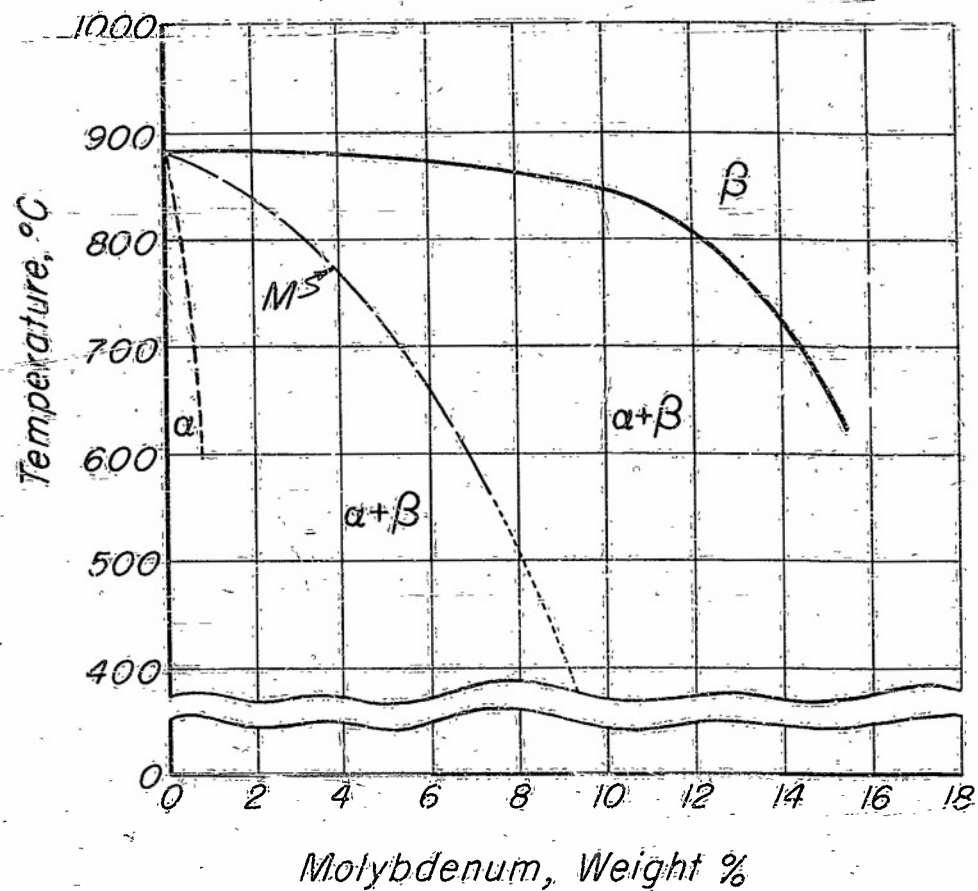Diagram (Metals Handbook 1948 Edition)

Fig. 7 — Titanium - Rich Portion of the Ti - Mo Equilibrium Diagram (Duwez)